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Enantiomorph-Polarity Estimation by Means of Flack's x Refinement: Practical Experiences

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Abstract

The refinement of 15 non-centrosymmetric crystal structures by means of Flack's x parameter is presented and compared with the standard practice of refining both possible coordinate sets separately. Particular emphasis is given to structures containing light to medium anomalous scatterers and to cases of inversion (merohedral) twinning. In all these cases, the results amply confirm x refinement to be efficient and physically meaningful. For inversion-twinned crystals in polar space groups where the origin may be freely chosen in at least one direction (*i.e.* those being subject to polar dispersion errors), it is shown in one example that only the proper treatment of twinning, *e.g.* by Flack's x parameter, results in unbiased atomic coordinates.

Introduction

In structure refinements of non-centrosymmetric crystals it is important to ensure that the atomic coordinate set of the model and the crystal have the same chirality or polarity. The ambiguities in the non-centrosymmetric crystallographic point groups thereby being resolved have been listed by Jones (1986*a*), who also coined the term 'absolute-structure determination' for the entire procedure (Jones, 1984*a*). In X-ray crystallography this may be effectively achieved by analysis of anomalous-dispersion effects.* In essence, this analysis consists of comparison of pairs of a reflexion and its Friedel opposite, preferably those which are most sensitive to anomalous-scattering effects (Bijvoet pairs). Common practice is to refine both a non-centrosymmetric structure and its inversion on a complete data set consisting of all unique reflexions and their Friedel opposites. The 'correct' absolute structure is expected to give a better fit to the observed data, as revealed by a discriminatory test (Hamilton, 1965; Pawley, 1970). Methods combining both steps utilize the simultaneous refinement of a parameter which unequivocally relates one absolute structure with its inversion. As first suggested by Rogers (1981) this may be done by a parameter η

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* The direct determination of the chirality/polarity of a crystal by measurement of triplet phase relationships has been described recently (Hümmer & Billy, 1986).

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which multiplies all the imaginary components f'' of the anomalous-dispersion terms of the atomic scattering factors. Despite the successful application of the η parameter (Jones, 1984*a*; Jones & Meyer-Bäse, 1987), Flack (1983) has pointed out that both from a theoretical and from a practical point of view there are some important shortcomings in η. These concerr primarily the treatment of reflexions not affected by anomalous scattering, the presence of weak anomalous scatterers, as well as the presence of inversion twinning and pseudosymmetry. He suggested an alternative method which consists of treating a non-centrosymmetric crystal as an inversion twin (Flack, 1983). Refinement of x, the fraction of one of the twin components, then not only gives a quantitative measure of the predominant twin domain, but should furthermore indicate the possible presence of enantiomorphic (merohedral) twinning. This is the more important, as inversion twinning may seriously bias atomic coordinates in polar space groups where the origin may be arbitrarily placed in at least one direction, *even if* anomalous scattering has been properly taken into account (Templeton, Templeton, Zalkin & Ruben, 1982, and references cited therein).

It should be noted at this point that refinement programs treating twinned crystals in a general manner have been developed and successfully applied to merohedral and pseudomerohedral twinning (Bärnighausen, 1985).

This paper reports experiences with Flack's x refinement with particular emphasis given to light-atom structures and inversion twinning.

Method

Data sets were measured either at 293 K on an Enraf–Nonius CAD-4 diffractometer or at 238 K on a Syntex $P2_1$ diffractometer. Graphite-monochromatized Mo $K\alpha$ radiation was used in all cases ($\lambda = 0{\cdot}71069$ Å). Table 1 contains some values pertinent to the range of data. Friedel opposites* were measured except for compounds (5) and (11). Further information on data collection and reduction procedures may be found in the individual publications. Absorption corrections were based on scans at intervals of 10° around the diffraction vectors of up to nine reflexions near $\chi = 90°$ (ψ scans) which served to evaluate the transmissions. The 'unobserved' threshold was set at $4{\cdot}0\sigma(F)$ with the exception of structure (15) where it was $2{\cdot}0\sigma(F)$. Unobserved data were not used in the refinements. The function minimized was $\sum w(|F_o| - |F_c|)^2$. It is acknowledged that refinement on $|F|^2$ would be preferable (Hirshfeld & Rabinovich, 1973; Bernardinelli & Flack, 1985) but unfortunately our programs have not yet been adapted to this. Refinements of the structures and their inverses were first carried out using *SHELX*76 (Sheldrick, 1976) with dispersion-corrected scattering factors except for H. The results of the refinements which give the best fit to the measured data are the ones reported in the individual papers. Table 1 contains the wR values for both refinements. It should be mentioned that H atoms were included in the structure-factor calculations of all compounds but were usually not refined. At a later stage, refinement of the parameter sets thus obtained was repeated including refinement of Flack's x parameter using *GFMLX*.* Statistical weights $[1/\sigma^2(F_o)]$ were used throughout the latter refinements. f' and f'' corrections were applied to the three heaviest atoms of each structure. The resulting wR and x values are included in Table 1.

Results and discussion

The enantiopole refinements of structures (1) to (9), *i.e.* those containing medium to heavy anomalous scatterers, amply confirm the findings of Bernardinelli & Flack (1985) that the x parameter provides a powerful method for the determination of the chirality or polarity of crystal structures. In general, the x parameter converged rapidly and stably, and convergence was independent of the starting point (usually 0·5). Actually its behaviour was much like a scale factor; one cycle of refinement was usually sufficient and dampening was never necessary. In cases where no inversion twinning was observed [structures (1), (3), (6), (7), (9)], the R, wR and coordinate values are virtually identical with the better result of conventional separate refinements of one structure and its inverse, provided that similar weighting schemes were used. Furthermore, in these cases x refined within its error margins to 0·0, thereby indicating agreement between refined structure and data set. The e.s.d.'s of x are generally small, thus excluding pseudosymmetry (Flack, 1983). It should be mentioned at this point that all compounds except (7) and (13) are synthetic materials made in achiral environments and from achiral starting materials. Hence, where no inversion twinning is observed these structures are the result of spontaneous (Pasteur) resolution upon crystallization. Information regarding their chirality or polarity was not available beforehand and, in fact, most of the substances are achiral in solution, *i.e.* they either racemize rapidly in solution or they adopt a chiral conformation only in the solid state. Therefore, the unambiguous refinement of chiral (7),

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* Strictly speaking, the term 'Friedel pair' denotes a reflexion – Miller indices *h,k,l* – and its antireflexion *–h,–k,–l*. Therefore, most of the data sets of Table 1 include equivalents of Friedel opposites. The distinction may be important in a discussion of systematic effects such as absorption or extinction. For simplicity, throughout this paper the term 'Friedel opposite' denotes both antireflexions and their equivalents.

* *GFMLX*, a highly modified version of *ORFLSD*, by Hirshfeld, Coppens, Leiserowitz & Rabinovich, with enantiopole refinement as incorporated by Flack (1984).

Table 1. *Some experimental details of non-centrosymmetric structure refinements*

Reference		Chemical formula	Space group	μ(Mo $K\alpha$) (mm^{-1})	$[(\sin\theta)/\lambda]_{\max}$ (Å^{-1})	*hkl* range	$R_{\rm int}$*	*wR* values† *A*	*B*	*C*	*x*	Notes
(1)	MECK	$C_{14}O_{12}Re_2$	$P2_1$	13·81	0·636	$+h,\pm k,\pm l$	—	0·031	0·044	0·031	0·00 (2)	(*a*),(*c*)
(2)	WOWO	$C_{26}H_{40}NO_4PSi_2W_2$	$P4_32_12$	7·47	0·639	$+h,+k,\pm l$	0·020	0·023	0·024	0·020	0·46 (1)	(*a*),(*c*)
(3)	WOSE	$C_{20}H_{26}OSeW$	$P2_12_12_1$	7·30	0·648	$\pm h,+k,+l$	—	0·033	0·049	0·023	0·01 (1)	(*a*),(*c*),(*d*)
(4)	FISE	$C_{20}H_{23}NSe$	$P2_12_12_1$	2·03	0·594	$+h,+k,\pm l$	—	0·035	0·034	0·027	0·52 (2)	(*a*),(*c*)
(5)	KAGE	$C_{14}H_{36}GeP_6$	$Pna2_1$	1·65	0·572	$\pm h,+k,+l$	0·021	0·030	0·032	0·028	0·35 (3)	(*b*),(*c*),(*e*)
(6)	AGBU	$C_{16}H_{16}AgCl_4Ga$	$P2_12_12_1$	3·09	0·595	$+h,+k,\pm l$	—	0·037	0·046	0·037	0·03 (3)	(*b*),(*c*)
(7)	ZNAS	$C_4H_6ClNO_4Zn$	$P2_12_12_1$	3·84	0·593	$\pm h,+k,+l$	—	0·026	0·045	0·026	0·01 (2)	(*a*),(*c*)
(8)	SNAP	$C_{14}H_{36}P_6Sn$	$P2_1$	1·41	0·593	$+h,\pm k,\pm l$	—	0·0430	0·0433	0·0412	0·48 (3)	(*b*),(*c*),(*f*)
(9)	OPAL	$C_{16}H_{18}CrO_9$	$P2_12_12_1$	0·67	0·594	$+h,\pm k,\pm l$	0·016	0·032	0·042	0·032	0·00 (2)	(*b*)
(10)	CLIP	$C_{24}H_{64}Li_2N_2P_2Si_4$	$P2_12_12_1$	0·25	0·594	$+h,+k,\pm l$	—	0·0392	0·0393	0·0396	0·09 (19)	(*a*)
(11)	ALLI	$C_{16}H_{41}AlLiN_2OP$	$P2_1$	0·16	0·617	$+h,+k,\pm l$	—	0·0521	0·0522	0·0521	0·18 (31)	(*b*),(*g*)
(12)	SIWI	$C_{15}H_{36}Si_3$	$P2_12_12_1$	0·22	0·572	$+h,+k,\pm l$	—	0·0526	0·0525	0·0527	0·94 (28)	(*b*)
(13)	MGAS	$C_4H_{11}MgNO_7$	$P2_12_12_1$	0·20	0·648	$\pm h,\pm k,+l$	0·015	0·0413	0·0415	0·0417	−0·64 (37)	(*a*)
(14)	HOM5	$C_{13}H_{18}O_4$	$P4_12_12$	0·09	0·615	$+h,+k,+l$	—	0·0361	0·0361	0·0367	−0·6 (38)	(*a*)
(15)	BUBA	$C_{15}H_{14}$	$P2_12_12_1$	0·06	0·594	$\pm h,+k,+l$	—	0·058	0·058	0·037	24 (12)	(*b*),(*d*)

Compounds are arranged in descending order of largest f''.

References: MECK: Beck, Schweiger & Müller (1987); WOWO: Malisch, Niecke, Güth, Schemm & Müller (1988); WOSE: Fischer, Gerbing, Müller & Alt (1987); FISE: Fischer, Tiriliomis, Gerbing, Huber & Müller (1987); KAGE: Karsch, Deubelly, Hanika, Riede & Müller (1988); AGBU: Schmidbaur, Bublak, Huber, Reber & Müller (1986); ZNAS: Schmidbaur, Bach, Riede, Müller, Helbig & Hopf (1987); SNAP: Karsch, Appelt & Müller (1986); OPAL: Dötz, Popall, Müller & Ackermann (1986); CLIP: Karsch, Appelt, Deubelly & Müller (1987); ALLI: Karsch, Appelt & Müller (1985); SIWI: Wiberg, Wagner, Riede & Müller (1987); MGAS: Schmidbaur, Müller, Riede, Manninger & Helbig (1986); HOM5: Hofmann, Hoffmann, Reber & Müller (1987); BUBA: Schmidbaur, Bublak, Schier & Müller (1987).

Notes: (*a*) 293 K data; (*b*) 238 K data; (*c*) data corrected for absorption; (*d*) *wR* values *A* and *C* differ owing to different weighting schemes used; *R* values and atomic coordinates were not significantly different, however; (*e*) inadvertently, instead of Friedel opposites, another equivalent data set was measured; refinements were done on the merged data set, which therefore is expected to be more precise; (*f*) Sn at 0, 0·5, 0; (*g*) Friedel opposites not measured.

* $R_{\rm int} = [\sum |F - \langle F\rangle| / \sum F]$; only given if more than one unique set of reflexions *and* their Friedel opposites were measured.

† $wR = [\sum w(|F_o| - |F_c|)^2/\sum wF_o^2]^{1/2}$; the *A* values refer to the coordinate set which was subsequently refined including Flack's *x* refinement; the *B* values to the inverse set, or, where necessary, to refinement in the enantiomorphous space group [(2) and (14)]; *C*: final *wR* after refinement including Flack's *x* parameter.

Table 2. *Intramolecular distances* (Å) *in* (5) *most affected by the polar dispersion error*

The three values result from refinement without taking into account inversion twinning (*A*; *B* = inverse coordinate set) and from refinement including Flack's *x* parameter (*C*). Column *D* lists the angles (°) between the respective bond vectors and the polar *z* axis. Note that the smallest discrepancies in the bond lengths arise for the bonds having the angle closest to 90° to the polar axis.

	A	*B*	*C*	*D*
Ge–P(1)	2·345 (2)	2·384 (2)	2·359 (2)	41·1
Ge–P(2)	2·551 (2)	2·535 (2)	2·546 (2)	77·2
Ge–P(4)	2·378 (2)	2·348 (2)	2·368 (2)	55·4
Ge–P(5)	2·920 (2)	2·936 (2)	2·926 (2)	71·1

ZnCl (L-aspartate) (Schmidbaur, Bach, Riede, Müller, Helbig & Hopf, 1987) to the expected absolute configuration serves as a valuable check that our experimental setup and the treatment of anomalous scattering in our programs determine absolute structures correctly.

Compounds (10) to (15) contain only light (usually from the second period of the Periodic Table) or no anomalous scatterers. The e.s.d.'s of *x* are consequently much larger, and exclude physically unmeaningful *x* values [see, for example (13)–(15)] from being significant. *x* refinement was stable in all cases although various starting points were tried out (0·0, 0·5, 1·0). Again, the *wR* values with and without refinement of *x* do not differ significantly. A particularly interesting case is compound (12), where the tiny difference in *wR* in the original refinements (Table 1) was not considered significant to favour the model with the smaller *wR* (Wiberg, Wagner, Riede & Müller, 1987). *x* refinement now indicates [$x = 0·94$ (28)] that the model with the smaller *wR* value is probably the correct one, although the rather large e.s.d. quantifiably does not give much weight to it.* Compound (13), an Mg complex of L-aspartic acid (Schmidbaur, Müller, Riede, Manninger & Helbig, 1986), shows that in this case the presence of magnesium clearly is not enough to allow a determination of the absolute configuration (with Mo $K\alpha$ radiation) although the *wR* values show small differences.

Particularly interesting are the cases [(2), (4), (5), (8)] where *x* refinement indicates the presence of inversion twinning. Again, the small e.s.d.'s of *x* exclude pseudosymmetry. As is clearly seen from the noticeably different *wR* values, the treatment of the twinning allows a better fit of the data, although in non-centrosymmetric space groups with fixed origin, *e.g.* $P2_12_12_1$, no first-order errors in the coordinates are to be expected (Cruickshank & McDonald, 1967). While

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* As described in the original publication (Wiberg, Wagner, Riede & Müller, 1987), there were no differences between the bond distances and angles calculated from the two refined models of opposite chirality. The geometry of the published structure is thus correct in all details other than its chirality.

for (8) no significant differences in the derived bond distances and angles were observed after the three different refinements (Karsch, Appelt & Müller, 1986; Müller, 1987), for (5) significant differences resulted from shifts of the P atoms along the z axis with respect to the fixed Ge atom (space group $Pna2_1$). These are summarized in Table 2. The x parameter indicates an approximate 1/3 to 2/3 ratio of the twin components which also explains the differences in the wR values in the two refinements without consideration of the twinning. Thus in this case the twinning could not even be deduced from about equal wR values in the customary refinement of the two alternative structures, as was possible for the 50:50 twins of (2) and (4)! Quite noticeably, the final bond distances and angles are about 1/3 between the extremes of the refinements without consideration of twinning.

Concluding remarks

Although the polar dispersion error resulting from neglect or wrong treatment of anomalous dispersion in polar non-centrosymmetric space groups has been pointed out long ago (Ueki, Zalkin & Templeton, 1966; Cruickshank & McDonald, 1967), recent evidence indicates that in a majority of cases such crystal structures are still not properly dealt with (Rogers & Allen, 1979; Jones, 1984*b*, 1986*b*). Experience in this laboratory indicates that some 10–15% of synthetic achiral molecules crystallize in non-centrosymmetric space groups, and 2–3% do so under inversion twinning. In addition to the plea of Bernardinelli & Flack (1985) for a suitable routine determination of the chirality/polarity of non-centrosymmetric crystal structures, it should be emphasized that a check on inversion twinning is likewise mandatory if unbiased atomic coordinates are to be obtained (Templeton, Templeton, Zalkin & Ruben, 1982, and references cited therein). Flack's x refinement provides an ideal means for the refinement of such structures including inversion twinning.

Experiences with truncated data sets, especially those not containing Friedel opposites, indicate that enantiopole refinement is rendered only slightly less efficient in these cases (Bernardinelli & Flack, 1987; Flack, 1988). On the other hand, refinement of non-centrosymmetric crystal structures containing very light or no anomalous scatterers [*e.g.* compounds (14) and (15)] on data sets containing Friedel opposites have reflexion-to-parameter ratios twice as good as 'traditional' refinements on limited data sets without them. Work is under way to find precise criteria as to when refinement including Friedel opposites becomes obsolete for light-atom non-centrosymmetric crystal structures.

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